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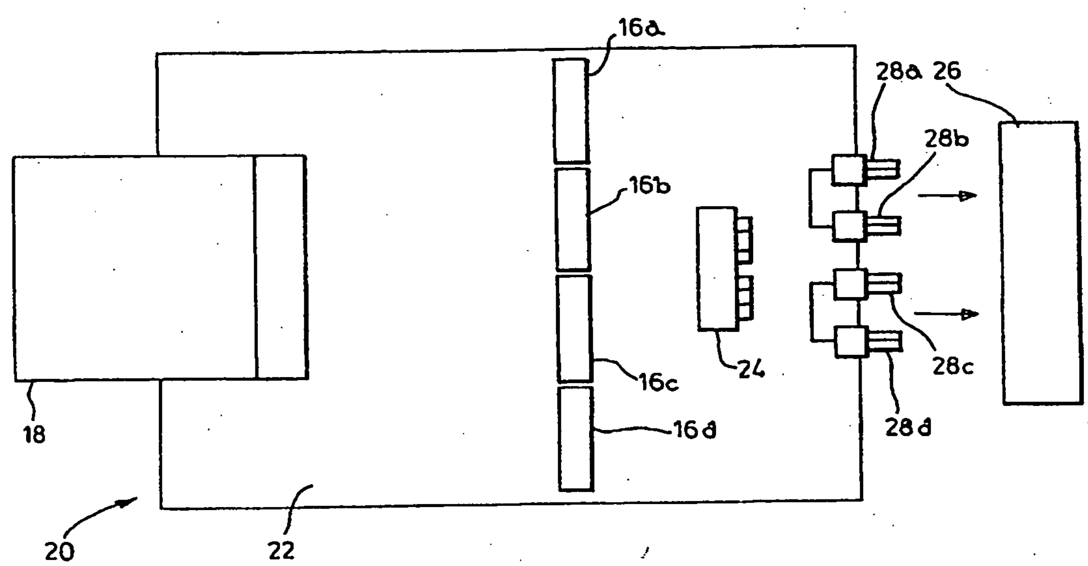

(57) Abstract

There is disclosed a composite gas sensor comprising a plurality of individual gas sensors electrically connected in a series or parallel arrangement, or in a combination of series and parallel arrangements.

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GAS SENSOR

This invention relates to gas sensors, in particular to a plurality of gas sensors electrically connected in series and / or parallel.

The use of semiconducting organic polymers (SOPs) as the active sensing medium in a variety of gas sensors is well established. Typically, a sensor comprises a pair of electrodes bridged by at least one layer of SOP; transduction is usually accomplished by measuring changes in the dc resistance of the sensors, these changes being induced by adsorption of gaseous species onto the polymer (see, for example, Persaud K C, Bartlett J G and Pelosi P, in "Robots and Biological Systems : Towards a new bionics?", Eds. Darios P, Sandini G and Aebisher P, NATO ASI Series F : Computer and Systems Sciences 102 (1993) 579). When used in conjunction with dc transduction, SOPs are not selective : a given SOP is sensitive to a range of species, perhaps a series of homologues possessing a functional group to which the polymer is responsive. In order to achieve chemical selectivity, arrays of sensors (often 20 or more) are employed, wherein each sensor utilises a different SOP and the SOPs employed possess differing but broadly overlapping sensitivities towards a range of molecules. Selective recognition of a given species is thus possible, since the pattern of sensor response across the array represents a characteristic molecular "fingerprint".

British Patent GB 2 203 553 discloses an improved method of sensor transduction wherein an ac electric signal is applied to a single sensor, and variations in an impedance characteristic, such as resistance, reactance or capacitance, are measured

as a function of ac frequency. One advantage of this approach is the increase in the information derived from a single sensor : in contrast to a single measurement made with the dc transduction technique, a plurality of measurements is made (at a variety of ac frequencies). In this instance, the spectrum of impedance characteristic variation against ac frequency provides the species specific "fingerprint", with the promise that a single sensor may be employed in place of an array of sensors comprising different SOPs.

The sensor configurations previously employed exhibit resonant characteristics : in fact, this property has proved to be an important aspect of gas sensing with ac transduction, since the impedance characteristics of the sensors give rise to high Q factors around the resonant frequency (Amrani, M E H; Persaud K C; and Payne, P A; Meas. Sci. Technol., 6 (1995) 1500). Furthermore, the amplitude and frequency of these high Q resonant peaks vary as sensitive functions of gas composition and concentration. However, the resonant frequencies of such prior art sensors are rather high; typically, resonant frequencies are ca. 600 MHz or greater. These high resonant frequencies are a disadvantage, because many instrument design problems are encountered in ac circuits which require applied ac frequencies in excess of ca. 10 MHz.

It would be highly desirable to provide a sensor having a resonant frequency of less than ca. 1 MHz, since instrumentation difficulties are much reduced at these lower frequencies. Furthermore, it would be desirable to reduce sensor to sensor response variations, which affect the accuracy and reproducibility of measurements made using either of the above described transduction techniques. Further still, it would be desirable to provide a convenient method for varying the resonant frequency. The present invention addresses these problems. It should be noted that 'gas' is understood to

include any gas phase species, including vapours. Although the invention is described herein with reference to ac and dc interrogation / transduction techniques, other techniques suitable for performing these functions are within the scope of the invention, such as the time to frequency domain interrogation technique described in International Publication WO 97/18467.

According to the invention there is provided a composite gas sensor comprising a plurality of individual gas sensors electrically connected in a series or parallel arrangement, or in a combination of series and parallel arrangements. The individual gas sensors may comprise a pair of electrodes and at least one layer of semiconducting organic polymer deposited onto and between said electrodes.

Preferably at least eight gas sensors are connected, most preferably at least thirty two.

A direct current electric signal may be applied to the composite gas sensor and variations in the resistance of the composite gas sensor detected. The use of a plurality of sensors results in an increase in sensitivity compared to a single gas sensor of the same type, and, very importantly, reduces the effect of sensor to sensor fluctuations in response characteristics due to statistical averaging of the sensor responses.

Alternatively, an impedance characteristic or a variation in an impedance characteristic of the composite gas sensor may be detected. The impedance characteristic

or the variation in the impedance characteristic may be detected as a function of frequency. An alternating electric signal may be applied to the composite gas sensor.

In addition to the above described advantages of sensitivity and statistical averaging of sensor responses, further advantages in this instance are that the resonant frequency of the sensor is controllable and stable.

The resonant frequency of the composite gas sensor may be less than 2MHz.

The composite gas sensor may comprise a silicon chip carrier. Said chip carrier may comprise a 64 pin chip carrier.

A plurality of composite gas sensors may be fabricated on a single substrate. For example, the silicon chip carrier may comprise a plurality of composite gas sensors - a 64 pin chip carrier may comprise four composite gas sensors, each composite gas sensor utilising a different SOP or SOP combination and comprising eight individual sensors connected in series.

The composite gas sensor may comprise a plurality of individual gas sensors electrically coupled to switching means, said switching means being operable to connect the individual gas sensors, or a subset thereof, in a series or parallel arrangement, or in a combination of series and parallel arrangements.

A composite gas sensor according to the invention will now be described with reference to the accompanying drawings, in which :

Figure 1 is a diagrammatic plan view of a 64 pin chip carrier with connector;

Figure 2 is a diagrammatic plan view of the adaptor unit;

Figure 3 shows series-connected sensor group reactance versus applied ac frequency; and

Figure 4 shows parallel-connected sensor reactance versus applied ac frequency;

Figures 1 and 2 show a composite gas sensor comprising a plurality of individual gas sensors 11 electrically connected in a series arrangement, or in a combination of series or parallel arrangements. Each individual gas sensor 11 comprises a pair of gold electrodes 14a, 14b and at least one layer of SOP 12 deposited onto and between said electrodes. There are many examples of suitable SOPs to be found in the literature (see, for example, International Application No. WO 86/01599); polypyrrole, polyaniline and derivatives thereof are common choices. Preferably there are at least eight gas sensors of the same type connected in series, most preferably - as depicted in Figure 1 - at least thirty two. It will be apparent to the skilled reader that other gas sensors might be employed which rely on the measurement of the electrical properties of a gas sensitive material, such as metal oxide (MOS) sensors.

Direct current electric signal may be applied to the composite gas sensor and variations in the resistance of the series-connected gas sensor may be detected.

Alternatively, alternating electric signal may be applied to the composite gas sensor and an impedance characteristic may be detected. Data obtained using this method of transduction are presented below.

It is found that the resonant frequency of the composite gas sensor is less than 2 MHz : in fact, one or two resonant frequencies are observed below ca. 1 MHz.

The composite gas sensor comprises a 64 pin silicon chip carrier 10. Figure 1 shows a plan view of the chip carrier 10 : polymer 12 is deposited onto and between thirty-two adjacent pairs of gold electrodes 14a, 14b producing what may be regarded as thirty two individual gas sensor 'units' 11. For the purposes of the present invention, these gas sensor 'units' are connected in series and / or in parallel.

The 32 individual gas sensors are divided into four separate groups of sensors, each group comprising eight individual gas sensors connected in series. Such is accomplished by use of four 12 way series connecting DIP switches 16a, 16b, 16c, 16d. Figures 1 and 2 depict the arrangement. The thirty-two sensor units 11 are connected via a 68 way ribbon cable connector 18 to an adaptor unit 20 fabricated on a Printed Circuit Board (PCB) 22. Since each sensor unit 11 comprises two electrodes, each group of eight sensors comprises sixteen connections. Each set of sixteen connections (not shown in figure 2) are input to separate series connecting DIP switches 16a, 16b, 16c, 16d, which are so configured to electrically connect the eight sensor units

of a group in series. Thus each series connecting DIP switch provides two outputs corresponding to a single sensor group.

The outputs of the four DIP switches 16a, 16b, 16c, 16d are electrically connected (connections not shown) to switching means 24, said switching means 24 being operable to connect four groups of sensors, or a subset thereof, in a series or parallel arrangement. The switching means 24 is a 12 way DIP switch.

As described above, one problem with prior art SOP based gas sensors is sensor to sensor variations in response caused by *inter alia* variations in manufacturing conditions and sensor ageing processes. The composite sensor of the present invention results in substantial averaging of these sensor to sensor variations. Uniformity of sensor response is of great importance in the field of commercial gas sensing devices (or "electronic noses") since it is desirable that sensor patterns obtained in the presence of a given gas (the molecular "fingerprint") are instrument independent. Such instrument independent patterns could then be regarded as an 'absolute' descriptor for the gas in question, and would permit cross-referencing or calibration with the outputs of other devices. In this instance, an electronic nose incorporating the present invention would consist of a plurality of arrays, each array comprising a number of gas sensors connected in series and / or in parallel. It should be noted that apart from reducing statistical fluctuations, the use of a number of gas sensors connected in series and / or parallel results in an increase in sensitivity (compared to an individual gas sensor) due to the increase in surface area of semiconducting organic polymer available for adsorption of gaseous species.

Further advantages still are found to accrue when the composite gas sensor of the present invention is used in conjunction with ac interrogation. For this purpose an impedance analyser 26 (Hewlett Packard 4192A) applies ac electric signal of variable frequency to the composite gas sensor and measures ac impedance characteristics thereof. The connection between the fixtures of the impedance analyser 26 and the adaptor unit 20 is made with four shielded BNC right angle sockets 28a, 28b, 28c, 28d, mounted on the PCB 22 and electrically connected to the switching means 24.

As described above, the thirty two individual gas sensors 11 are divided into four groups, hereinafter referred to as R1, R2, R3 and R4. Each group comprises eight individual gas sensors connected in series via the series connecting DIP switches 16a, 16b, 16c, 16d. The switching means 24 permits selection of any desired combination of groups Rl to R4, electrically connected in either a series or parallel arrangement. Measurements of reactance were made using the impedance analyser in the range 600 to 1200 KHz. Figure 3 (a) shows the reactances 30, 32, 34, 36 respectively corresponding to group R1, groups R1-R2 connected in series, R1-R2-R3 in series, and R1-R2-R3-R4 in series in the range 600 to 1200 KHz. In like manner, Figure 4 (a) shows reactances 40, 42, 44, 46, respectively corresponding to group R1, groups R1-R2 connected in parallel, R1-R2-R3 in parallel, and R1-R2-R3-R4 in parallel in the range 600 to 1200 KHz.

Figure 3 (a) shows that for the series sensor group arrangement the reactance reaches zero twice in the 600 to 1200 KHz range, indicating that the ac circuit involved exhibits two resonant frequencies. The lower resonant frequency appears between 700 to 760 KHz, whilst the higher resonant frequency appears around 1080

KHz. In the vicinity of the higher resonant frequency, the transition from positive to negative values of reactance is steep, with large values of $\left|\frac{dX}{df}\right|$. In contrast, this transition is rather shallow around the lower resonant frequency. As shown in Figure 4 (a), the parallel sensor group arrangement gives rise to a single resonant frequency around 1080 KHz. As expected, the magnitude of the measured reactance increases as more groups of sensors are connected in series, and decreases as more groups of sensors are connected in parallel.

Figures 3(b) and 4 (b) show reactance between 600 and 800 KHz - encompassing the lower resonant frequency - on an expanded scale for the series and parallel arrangements respectively. Similarly, Figures 3 (c) and 4 (c) show reactance between 1074 and 1090 KHz - encompassing the higher resonant frequency - on an expanded scale for the series and parallel arrangements respectively. The higher resonant frequencies are observed in a relatively narrow frequency range between 1079 and 1084 KHz. Nevertheless, the resonant frequency of a configuration is distinct from that of other configurations. Much wider variations are observed with the lower resonant frequencies shown in Figure 3 (b), which are spaced between 700 and 760 KHz. As shown in Figure 4 (b), connection of the groups of sensors in parallel arrangements appears to result in quite dramatic shifts in the lower resonant frequency to below 600 KHz. Features such as reactance, resonant frequency and dissipation factor may be utilised in the identification of gas samples using ac interrogation (see, for example, Amrani et al. ibid).

It is believed that the observed pattern of resonant frequency shifts can be explained by considering mutually induced phenomena such as mutual inductance and capacitance.

In a series arrangement of sensors, the leads connecting the sensors to the impedance analyser will have:

1) A self inductance L_S which is the inductance of the physical lead.

2) Mutual inductance M resulting from the flow of current in opposite directions for each adjacent pair of leads. This mutual inductance is differentially coupled and hence substracted from the total inductance L_T:

$$L_T = L_S - M$$

Thus the series configuration results in a decrease in inductance.

3) Mutual capacitance C_M, since adjacent leads have charges of opposite polarity.

Therefore there is an increase in lower resonant frequency due to L_T and C_M, and a small decrease in the higher resonant frequency due to the contribution of the total series capacitance of the sensing elements C_T which is more predominant at higher frequencies.

In a parallel arrangement of sensors, current flows through adjacent leads in the same direction, producing:

1) A self inductance L_S.

2) A mutual inductance M due to the currents travelling in the same direction for each adjacent pair of leads. This inductance is cummulatively coupled and hence added to the total inductance L_T :

$$L_T = L_S + M$$

Thus there is an increase in terms of total inductance.

3) There is no mutual capacitance as the current is travelling in the same direction.

Therefore there is a considerable decrease in resonant frequency in the lower part of the frequency range and a very small decrease at the higher resonant frequency due to the slope of the reactive characteristic of the device.

The resonant frequencies of prior art gas sensors employing semiconducting organic polymers and ac transduction are rather high; typically resonant frequencies are ca. 600 MHz or greater. It is well known that the problems of instrumentation design are greatly exacerbated when measurements are required at such high frequencies; and therefore it is an important facet of the present invention that the resonant frequencies may occur in a far more tractable frequency range below ca. 1 MHz. whilst high Q resonant behaviour is found to be retained. The nature of the connections to the impedance analyser have been found to play an important role. In particular, high

connector impedance results in lower resonant frequencies, whilst good connector dielectric properties produce the high Q resonant characteristics.

Connection of sensors or groups of sensors in parallel permits further adjustment of the resonant frequency. In this way, it is possible to substantially select the ac frequency range in which a device is to be operated, notwithstanding the physical characteristics of individual gas sensors.

It should be noted that a further advantage is that the measured resonant frequencies are very stable.

The use of a silicon chip carrier in the manufacture of SOP based gas sensors array is extremely convenient, although other forms of gas sensor arrays are within the scope of the invention. In the embodiment described above a thirty two sensor array was employed having identical individual gas sensors. Sets of eight individual gas sensors are connected in series, producing four sensor "groups" which are themselves connected in series or parallel arrangements. It is also possible to employ groups of sensors having different SOPs or SOP combinations. For example, four different SOP or SOP combinations may be deposited onto a 64 pin chip carrier. Identical individual gas sensors can be connected together to produce, for example, four separate and differing composite gas sensors "groups" on one chip carrier. In this instance, it is unlikely that the four composite gas sensors would themselves be mutually electrically connected.

Another possibility is to employ a switching device that permits selection of any combination or combinations of sensors in series and/or parallel arrangements. One application would be to "shut down" individual sensors which are malfunctioning or whose base resistance exceeds a predetermined value.

CLAIMS

1. A composite gas sensor comprising a plurality of individual gas sensors electrically connected in a series or parallel arrangement, or in a combination of series and parallel arrangements.

2. A composite gas sensor according to claim 1 in which the individual gas sensors comprise a pair of electrodes and at least one layer of semiconducting organic polymer deposited onto and between said electrodes.

3. A composite gas sensor according to claim 1 or claim 2 in which at least eight gas sensors are connected.

4. A composite gas sensor according to claim 3 in which at least thirty two gas sensors are connected.

5. A composite gas sensor according to any of claims 1 to 4 in which direct current electric signal is applied to the composite gas sensor and variations in the resistance of the composite gas sensor are detected.

6. A composite gas sensor according to any of claims 1 to 4 in which an impedance characteristic or a variation in an impedance characteristic of the composite gas sensor is detected.

7. A composite gas sensor according to claim 6 in which the impedance characteristic on the variation in the impedance characteristic is detected as a function of frequency.

8. A composite gas sensor according to claim 6 or 7 in which an alternating electric signal is applied to the composite gas sensor.

9. A composite gas sensor according to any of claims 6 to 8 having a resonant frequency of less than 2MHz.

10. A composite gas sensor according to any of the previous claims comprising a silicon chip carrier.

11. A composite gas sensor according to claim 10 comprising a 64 pin chip carrier.

12. A plurality of composite gas sensors according to either claim 10 or 11 fabricated on a single substrate.

13. A composite gas sensor according to any of the previous claims comprising a plurality of individual gas sensors electrically coupled to switching means, said switching means being operable to connect the individual gas sensors, or a subset thereof, in a series or parallel arrangement, or in a combination of series and parallel arrangements.

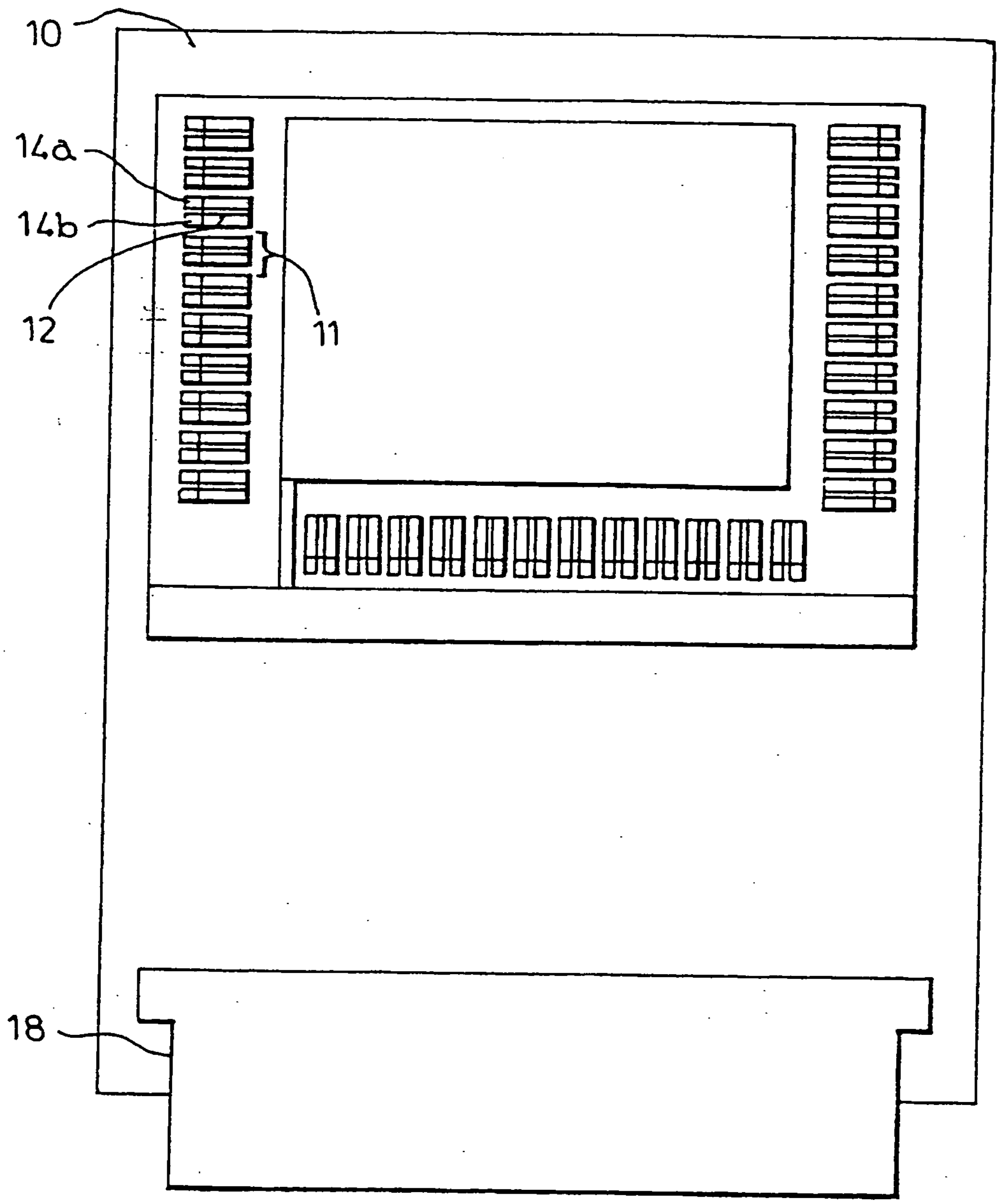


FIG.1

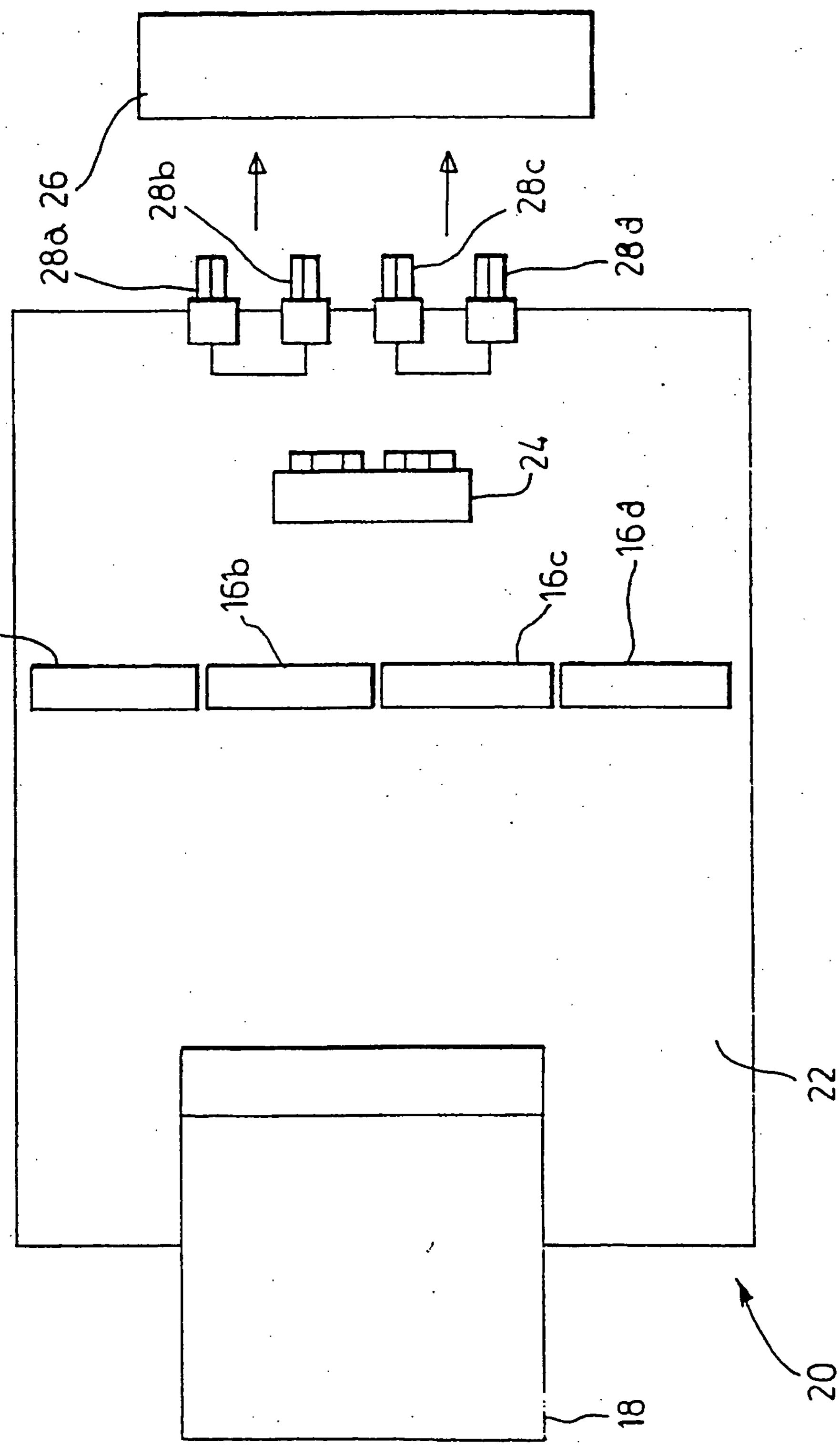
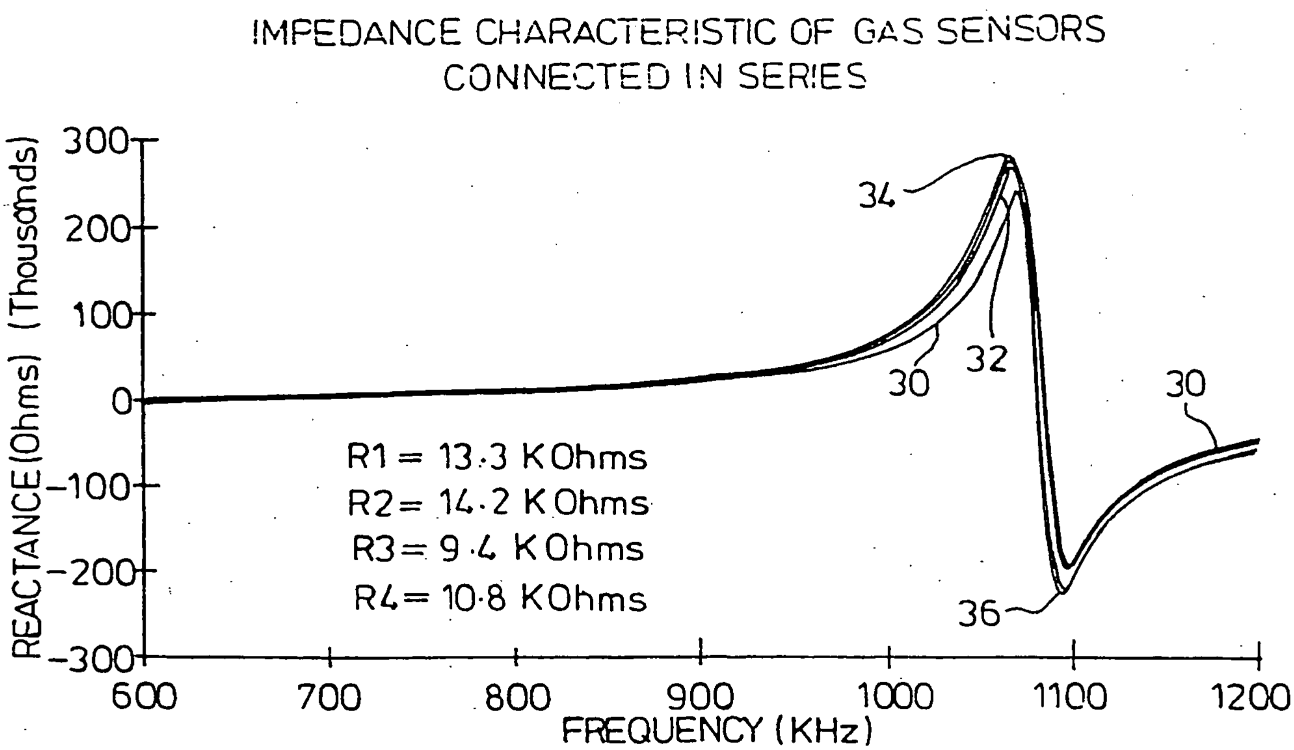


FIG.2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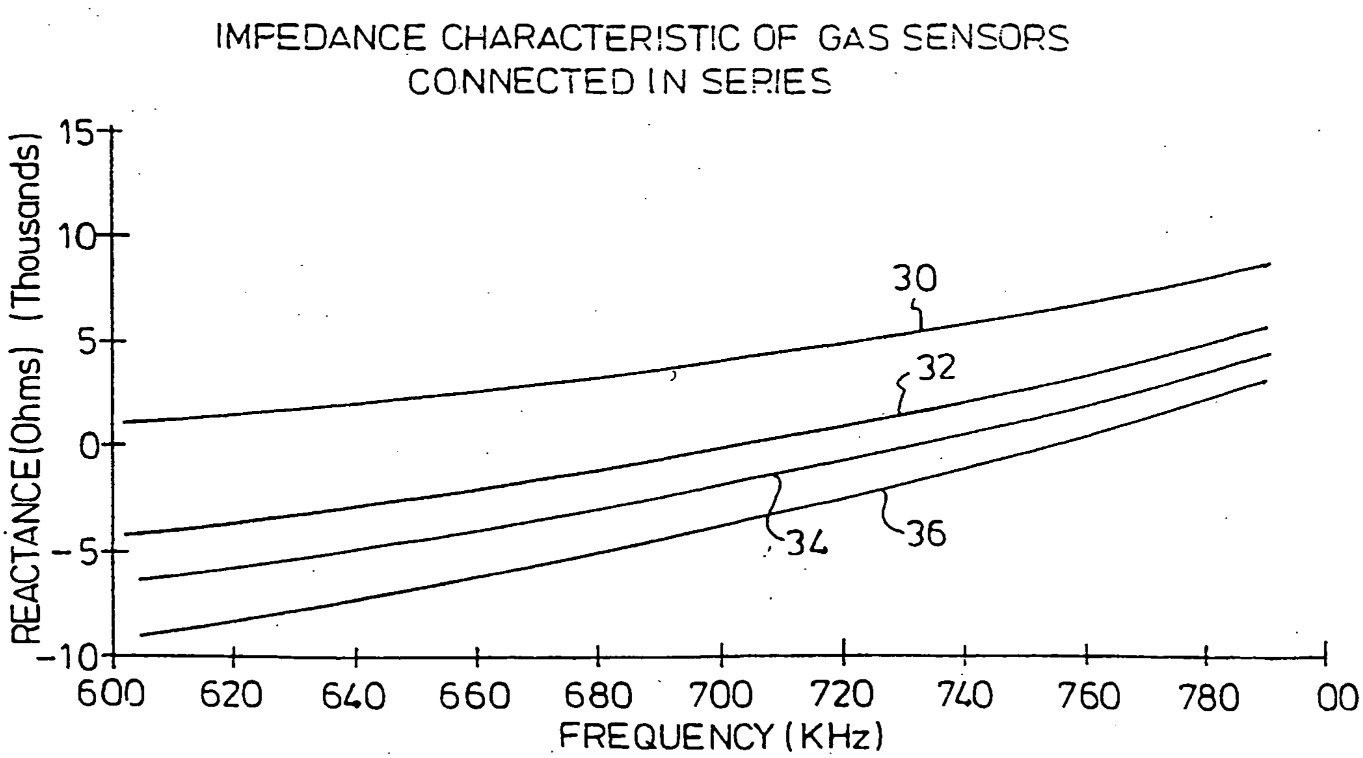
CONNECTED IN SERIES
REACTANCE (Ohms) (Thousands)
300
200
100
0
-100
-200
-300
600
700
800
900
1000
1100
1200
FREQUENCY (KHz)
R1 = 13.3 KOhms
R2 = 14.2 KOhms
R3 = 9.4 KOhms
R4 = 10.8 KOhms
34
32
30
30
36

FIG. 3. (a)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REACTANCE (Ohms) (Thousands)
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10
5
0
-5
-10
600
620
640
660
680
700
720
740
760
780
00
FREQUENCY (KHz)
30
32
34
36

FIG. 3 (b)

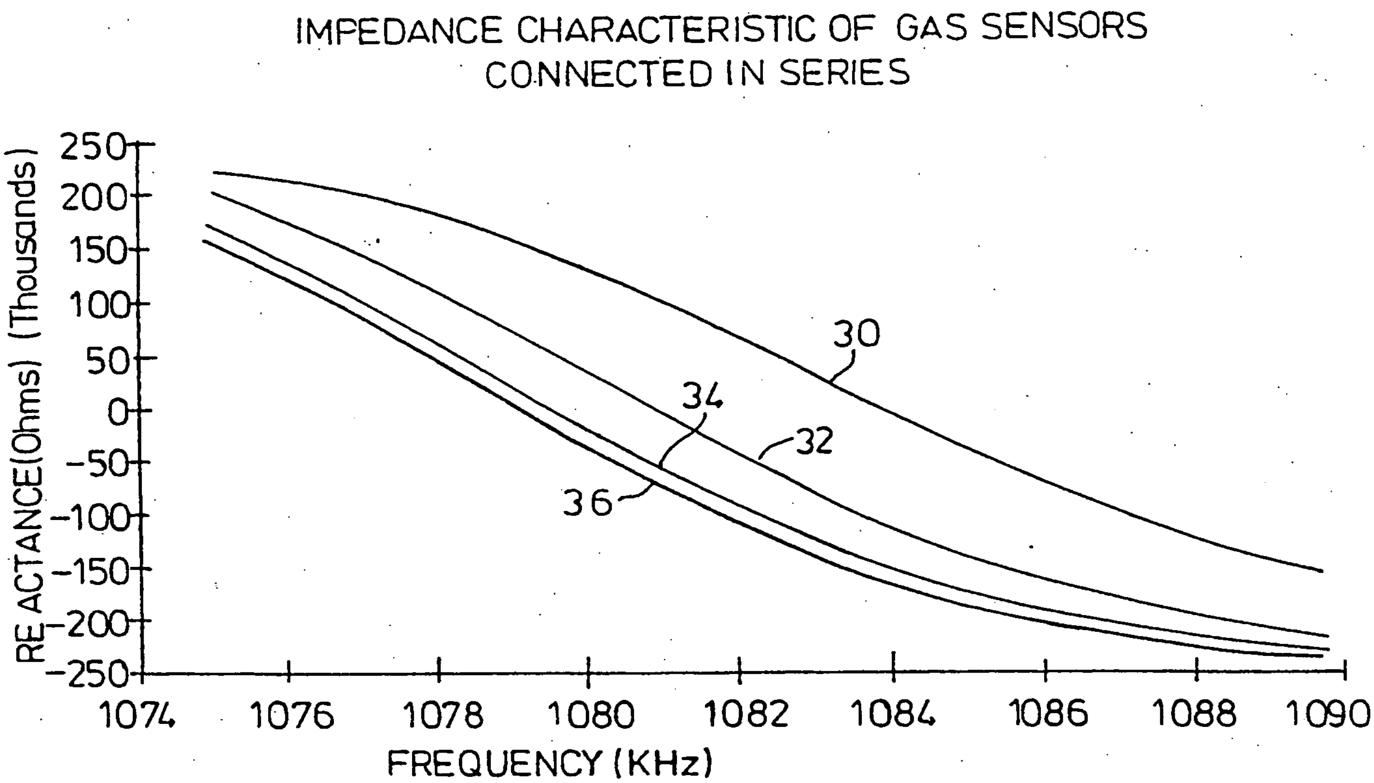

IMPEDANCE CHARACTERISTIC OF GAS SENSORS
CONNECTED IN SERIES
REACTANCE(Ohms) (Thousands)
250
200
150
100
50
0
-50
-100
-150
-200
-250
1074
1076
1078
1080
1082
1084
1086
1088
1090
FREQUENCY (KHz)
30
34
32
36

FIG.3(c)

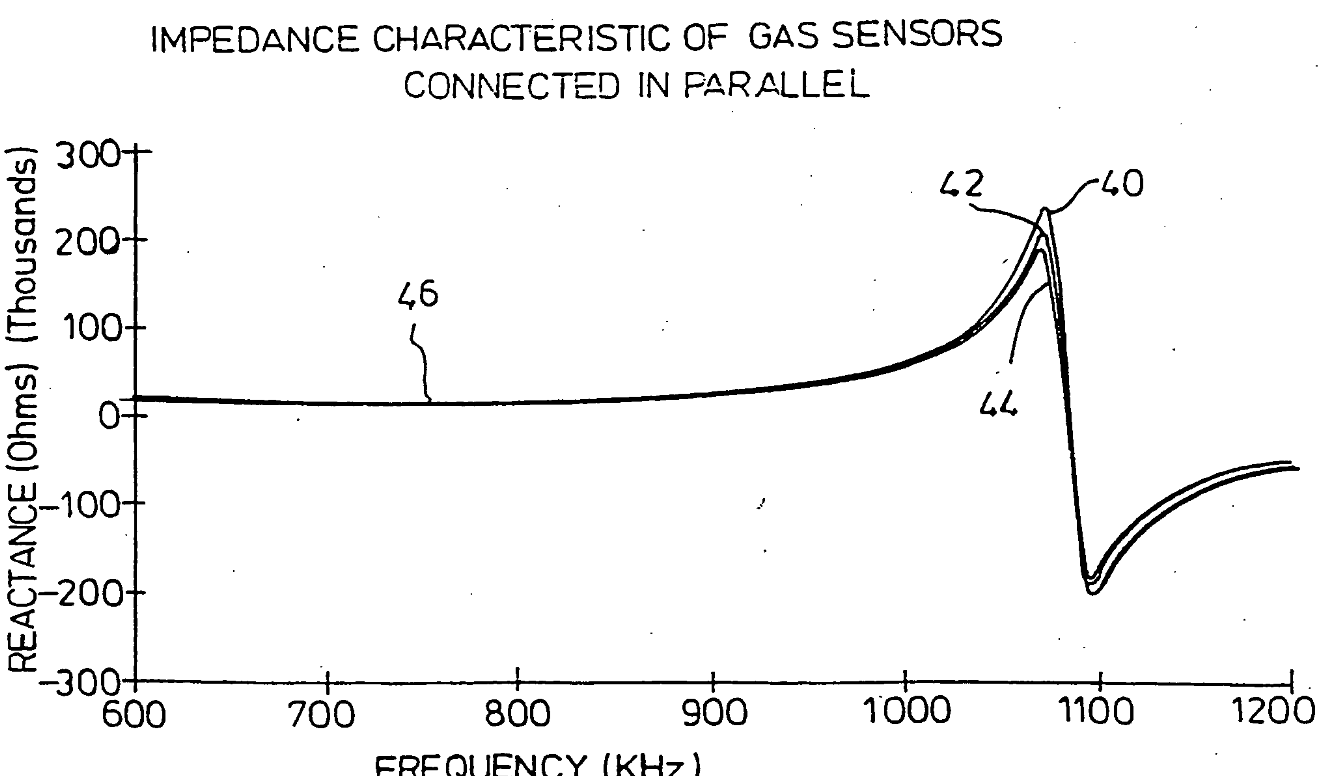
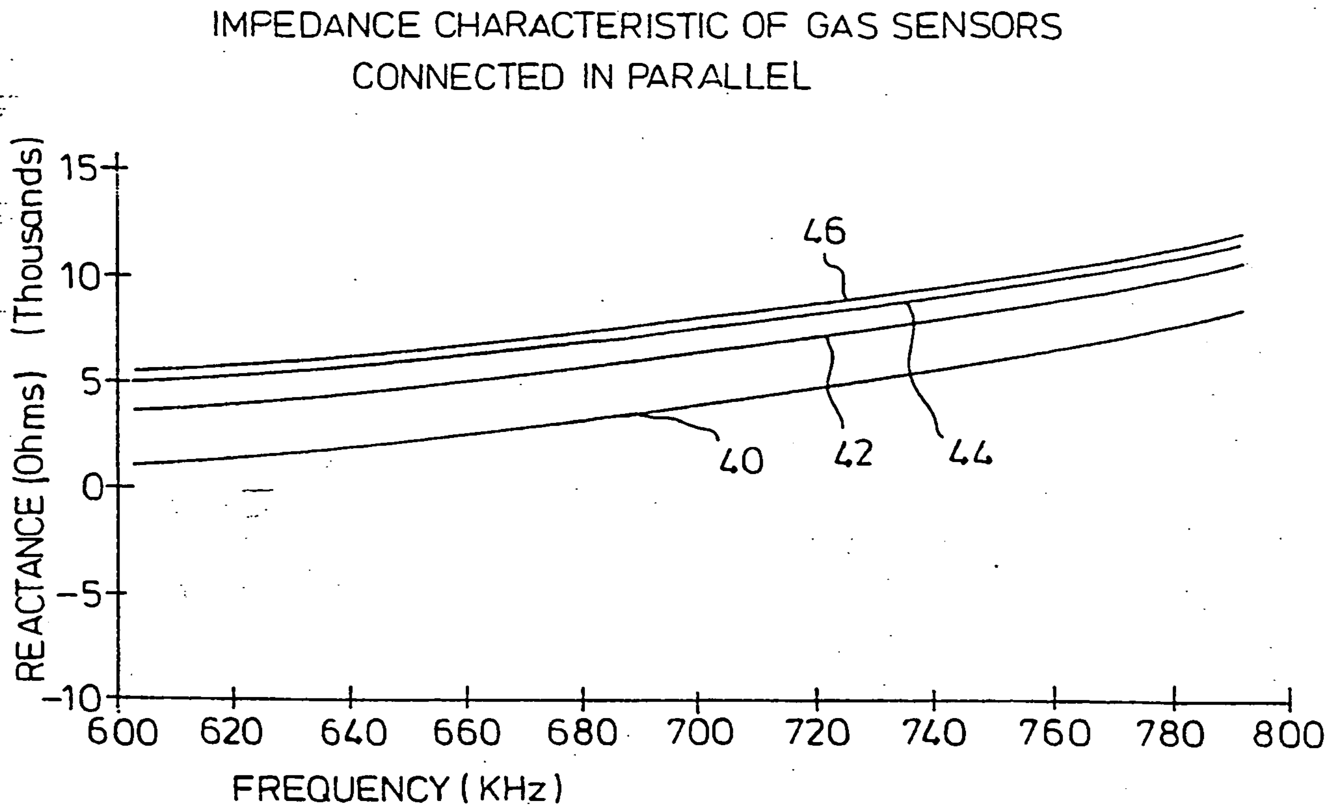
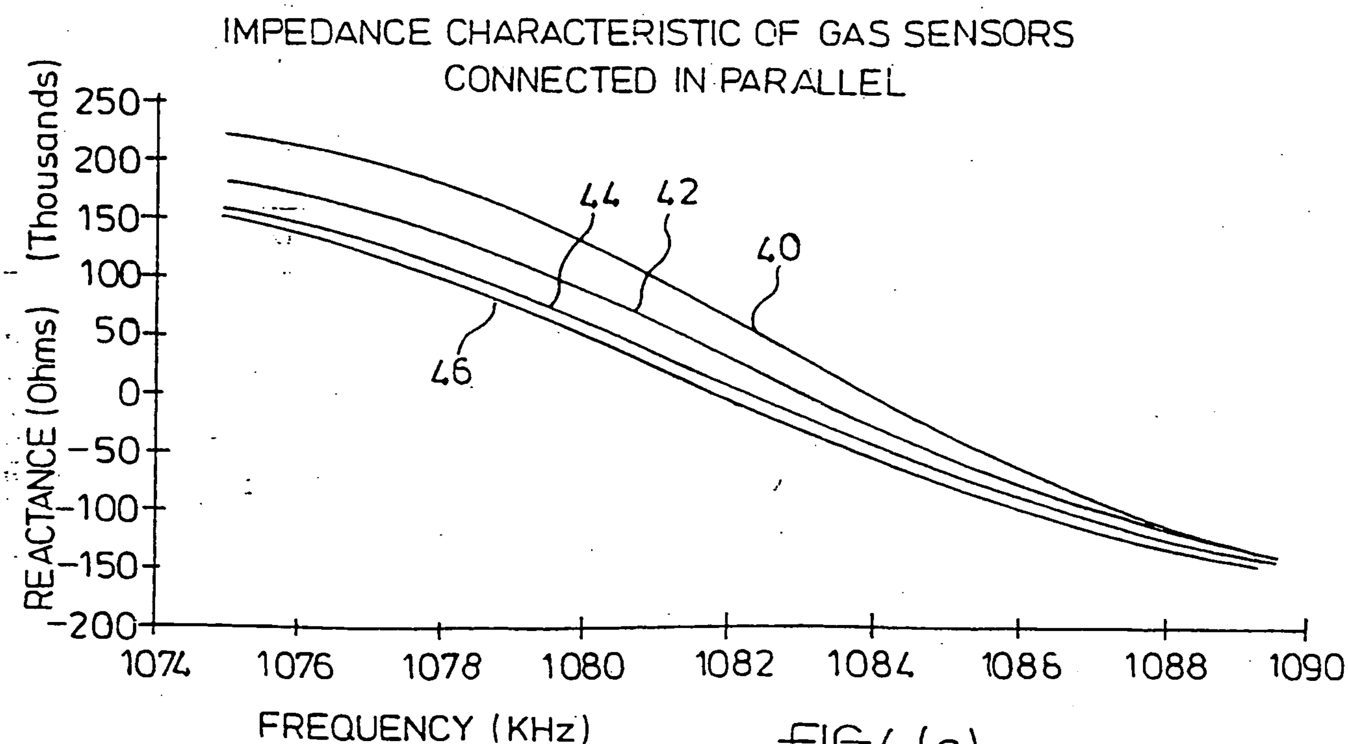

IMPEDANCE CHARACTERISTIC OF GAS SENSORS
CONNECTED IN PARALLEL
REACTANCE (Ohms) (Thousands)
300
200
100
0
-100
-200
-300
600
700
800
900
1000
1100
1200
FREQUENCY (KHz)
42
40
46
44

FIG.4 (a)



IMPEDANCE CHARACTERISTIC OF GAS SENSORS
CONNECTED IN PARALLEL
REACTANCE (Ohms) (Thousands)
15
10
5
0
-5
-10
600
620
640
660
680
700
720
740
760
780
800
FREQUENCY (KHz)
46
40
42
44

FIG.4 (b)



IMPEDANCE CHARACTERISTIC OF GAS SENSORS
CONNECTED IN PARALLEL
REACTANCE (Ohms) (Thousands)
250
200
150
100
50
0
-50
-100
-150
-200
1074
1076
1078
1080
1082
1084
1086
1088
1090
FREQUENCY (KHz)
44
42
40
46

FIG.4(c)

INTERNATIONAL SEARCH REPORT

Inte[illegible]onal Application No
PC[illegible] 98/01783

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 G01N33/00 G01N27/12

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	PATENT ABSTRACTS OF JAPAN vol. 9, no. 156 (P-368) '1879!, 29 June 1985 & JP 60 031049 A (MATSUSHITA DENKI KK), 16 February 1985	1,5,13
Y	see abstract	2-4,6-8, 10-12

Y	WO 96 00896 A (AROMASCAN PLC) 11 January 1996 see page 4, paragraph 6 - page 11, paragraph 2; figures	2-4,6-8

Y	WO 96 00384 A (AROMASCAN PLC) 4 January 1996 see page 5, paragraph 7 - page 10, paragraph 1; figures	10-12

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
7 October 1998	21/10/1998

Name and mailing address of the ISA	Authorized officer
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INTERNATIONAL SEARCH REPORT

Inter. onal Application No
/GB 98/01783

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 95 32422 A (AROMASCAN PLC) 30 November 1995 see the whole document	1,5
A	WO 96 30750 A (CALIFORNIA INSTITUTE OF TECHNOLOGY) 3 October 1996 see page 6, line 7 - page 31, line 16; figures 1-9	1-13
A	WO 93 08467 A (CAPTEUR SENSORS & ANALYSERS LTD.) 29 April 1993 see page 11, line 12 - page 17, line 8; figures	1,5
A	K.C. PERSAUD, ET AL.: "Design strategies for gas and odour sensors which mimic the olfactory system" ROBOTS AND BIOLOGICAL SYSTEMS: TOWARDS A NEW BIONICS? ED. BY PAOLO DARIO, ET AL., pages 579-602, XP002079909 Springer-Verlag, Berlin cited in the application see the whole document	1-13

1

INTERNATIONAL SEARCH REPORT

Inform[illegible]n patent family members

Inter[illegible]onal Application No
PC[illegible] 98/01783

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 9600896 A	11-01-1996	AU 2800995 A	25-01-1996
		EP 0767908 A	16-04-1997
		JP 10502172 T	24-02-1998
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